

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of:  
Arjun G. Yodh et al.

Confirmation No.: 7568

Application No.: 10/526,941

Group Art Unit: 1793

Filing Date: September 8, 2005

Examiner: Brittany M. Martinez

For: Carbon Nanotubes: High Solids Dispersions and Nematic Gels Thereof

Commissioner for Patents  
P.O. Box 1450  
Alexandria, VA 22313-1450

DECLARATION PURSUANT TO 37 C.F.R. § 1.131

I, Mohammad F. Islam, hereby declare that:

1. I am an inventor of the invention described and claimed in U.S. Patent Application Number 10/526,941 (hereinafter referred to as "the 941 application"), filed September 8, 2005, in the United States Patent and Trademark Office.
2. I am aware that the pending claims of the 941 application have been rejected as being unpatentable over U.S. Patent Application Pub. No. 2003/0133865 ("Smalley"). It has been explained to me that Smalley was filed on July 2, 2002, but that it claims priority from four U.S. provisional applications (collectively referred to as "the Smalley provisionals"):
  - Serial No. 60/303,469, filed July 6, 2001;
  - Serial No. 60/303,470, filed July 6, 2001;
  - Serial No. 60/337,561, filed November 8, 2001; and
  - Serial No. 60/337,951, filed December 7, 2001.
3. It has been explained to me that none of the Smalley provisionals disclose the use of at least one surfactant comprising an aromatic group.

4. In accordance with CFR § 1.131, as an inventor of the subject matter of the pending claims, and without conceding the propriety of the rejections of the pending claims, I hereby declare that I invented the subject matter with the inclusion of at least one surfactant comprising an aromatic group prior to July 2, 2002. I further hereby declare that I worked diligently from a date prior to July 2, 2002, to the date of constructive reduction to practice, September 10, 2002, the priority date of the 941 application, in order to prepare the 941 application and patent the invention.

5. In support of the instant declaration, a copy of relevant pages of a laboratory notebook prepared during the development of the claimed invention is attached hereto (Attachment B), which was created prior to July 2, 2002. The date range is from May 22, 2002 through July 20, 2002, which provides evidence of conception of the invention prior to the effective date of the Smalley reference and evidence of due diligence from prior to said date to the filing date of the provisional application, September 10, 2002.

6. I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information or belief are believed to be true; and further, that these statements were made with the knowledge that willful false statements and the like are punishable by fine or by imprisonment, or both, under § 1001 of Title 18 of the United States Code, and that such willful statements may jeopardize the validity of the application, any patent issuing thereupon, or any patent to which this verified statement is directed.

Date: 01/20/2010

Signature: M. F. Islam  
Mohammad F. Islam

## **Exhibit A**

### **Mohammad F. Islam**

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#### **EDUCATION**

2000 Ph.D. Physics, Lehigh University, Bethlehem, PA 18015  
1996 M.S. Physics, Lehigh University, Bethlehem, PA 18015  
1994 B.S. Physics,

#### **WORK EXPERIENCE**

2005-Present Associate Professor, Chemical Engineering and Materials Science and Engineering  
Carnegie Mellon University, Pittsburgh, PA 15213

2002-2005 Postdoctoral Fellow, Department of Physics and Astronomy  
University of Pennsylvania, Philadelphia, PA

#### **HONORS AND AWARDS**

2007 Alfred P. Sloan Research Fellow  
2007 National Science Foundation Career Award  
2006 American Chemical Society PRF Award  
1999 Sigma Xi  
1997 Hoechst Celanese Award for Excellence in Polymer Science

#### **CURRENT RESEARCH INTERESTS**

Novel Self-Assembly and Phase Transformations in Single- and Multi-Component Systems: Formation of diverse structures as well as phase transformations in multi-component systems using temperature sensitive colloidal particles.

Utilizing Nanomaterials to Investigate Cellular Functions: Developing novel nanomaterial based vectors and investigating changes in cellular functions due to internalization of these vectors.

Carbon Nanotube Based Porous Materials for Energy Applications: Created ultra-light, highly porous materials with carbon nanotubes; Investigating use as electrodes and support for catalyst particles.

Dependence of Cell Functions on Substrate Properties: Developed polymeric hydrogels with tunable local stiffness and organization; Using materials to probe dependence of cellular functions on substrate stiffness and spatial organization.

## Exhibit B

05/22/02

Make 2 samples of 1% Hipco + 10% NaDBS in water  
 each sample weighs 3g

add Hipco ~~0.02g~~

0.0321g

0.0324g

Make

~~add~~ NaDBS  
stock

2.002g

Total

10.03

} 19.96%

AddHipco  
NaDBS

0.0321g

1.5221g

0.0324g

1.5103g

water

1.4653g

1.4762g

Sample #3

Hipco

0.0314g

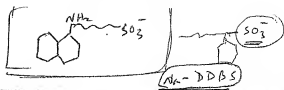
NaDBS

1.501g

water

1.480g

## Dioxy Cholate



### NT Project:

1. Scheme to separate & stabilize CNT without damaging it.

Tip Sonication (low f, high power) Better ultrasonication (high f)

- Advantage over pyrene functionalized CNT (damages CNT & ~~etc.~~ destroys electronic property)

- Comment on surfactant types for longer & better stability & separation

\* Benzene & charge surfactant best (NaDDBS)

(Literature is mostly on SDS)  
(one paper ODA)

2. Fractionate CNT using either HPLC, SEC

or GPC

↑  
Can use this

↑  
we use this

One paper on using GPC but do not elaborate & show careful results. (Duesberg's group)

- Can use this technique for other Rod shaped ~~or~~ particles.

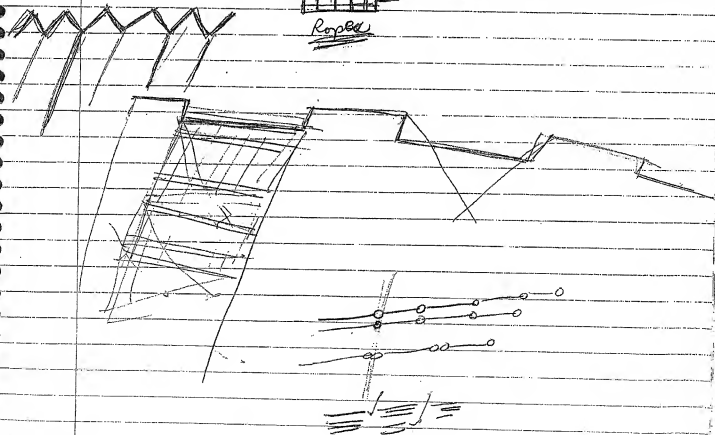
### 3. Characterization:

Use mainly LS  $\leftarrow$  large statistics

Cross check for a few functions w/  
AFM

### 4. Impact: stable, single CNT dispersions

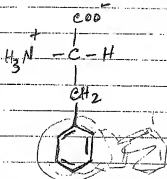
Assembly  $\rightarrow$  Controlled deposition . . .  
Rope



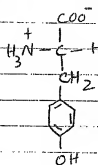
05/19/02

Bill Degrad

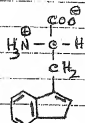
the more surfactants to try:



Phenyl alanine  
(F)  
Phe



Tyrosine



Tryptophan (W)  
[Trp]

or Trimer of F (F3)

Also try chaps & PEG

chiral proteins Bill Degrad JMB 2001

Butyl group, OH, benzene, amide group

Can we make chiral molecules for wrapping around  
Use Hydrogen bonding.

06/18/02

# Current Status of NT dispersion & characterization:

1% NT in NaDBBS

Bathsonic dispersion

bipsonic dispersion

electron  
phonons  
through  
0.5%  
Agarose  
gel

Run through  
Sephacrose 2B

AFM of  
sample as is

electrophoresis  
through 0.5%  
Agarose  
gel

AFM of  
sample as is

cut gel  
into  
small  
segments

sample  
comes through  
the gel.  
some of the  
solution gets  
trapped into

Unsuccessful;  
sample does  
not stick  
to surface

most did not  
go through;  
seems like not  
a good technique  
to disperse HPCs

unsuccessful;  
sample does  
not stick  
to surface

modify surface

Abandon this  
method

ultra melt  
cathode  
gel  
through  
fitter  
the  
most  
HPCs  
get  
stuck  
in  
fitter  
good  
tech  
high  
abundant

trapped into  
the gel  
(long elution  
time)  
They are  
presumably  
amorphous  
Carbon,  
bulky ball  
etc

saline  
similar  
as poly-L-L

check  
surface quality  
& see if NT  
sticks to the  
surface

poly-L-L  
successful;

sample  
HPCs stick to  
surface after  
dipping into soln  
of HPCs

next step  
check the surface  
quality & see  
how NT looks like

collect  
sample in  
multiple  
vials.

next step

look at a few  
of them to see  
if how good is the  
separation

Try this  
approach  
more



07/01/2002

50 wt  
of sample  
placed  
on chip  
was for  
5 min  
shaken off

MI 10-4.000

Silane treated  $10^{-4}$  H<sub>2</sub>O 5<sup>th</sup> scan in NAD<sub>2</sub>  
(Too much surf)

MI 10-6.001

" "  $10^{-6}$  " "  
(Too much surf/diluent)

MI 10-6.002

diff area, same chip

→ 10-4.003

$10^{-4}$  chip rinsed in H<sub>2</sub>O ~2min

10-4.004

diff area, same chip

10-4.005

$10^{-4}$  prepared by dunking in soln &  
then rinsing with water, dried  
with canned air

07/08/2002

Prepare 4g of 0.01% NT in NaDBS from 1% NT in NaDBS

1% NT soln

~~0.04g~~

0.0443g

} 0.01%

water

4.4434g

07/08/02

AFM on NaDBS stabilized HPCO

ddb510-4.000

zoom on the surface, leave it on  
for 10 min, spin it at 3000 rpm  
then add 2ml of water and the  
sample is spinning.

→ no feature

ddb510-4.001

same sample - diff. location

ddb510-4.002

dipped in soln, ~~then~~ for  
5 min, then gently rinse  
it in a water bath  
- lots of NT

ddb510-4.003

zoomed in some where  
in the previous ~~sample~~ picture

.004

different place on sample (SP)

.005

Zoom in of .004 (SP)

.006

new location 10μ (SP)

ddb510-4.007

12<sup>th</sup> cut of gel ~~ph~~  
phosais sample

cone ~ 10<sup>-4</sup> NT in ddb5

I dipped the chip  
in soln & then dried  
& then rinsed in  
water.

07/11/02

what I need to do:

conc

SDS

Tx100

NaDDBS

Prepare 1% → dilute to 0.01%

↑  
do not sonicate

↑  
just dilution

Prepare 0.01% sample

✓✓

✓

↑  
sonicate

I want to make 10% HiPCO soln.

9.96%

→ HiPCO 0.0009g

Add Rest 20% NaDDBS to get 2.009g

→ Final wt 2.0061g

07/12/02

Make 20 wt% TX-100 stock soln (50 grams)

TX-100 add 10 g  $\frac{10.00 \pm 0.01 \text{ g}}$

Rest add water till 50g Total 50.00 ~~24~~ g

Make 49% 1% TX-100 AdT soln w/ TX-100 (10%)

$$\left. \begin{array}{l} \text{NT} = 0.0397 \text{ g} \\ \text{TX-100} = 2.0085 \text{ g} \\ \text{(20 wt\%)} \\ \text{water} = 1.9913 \end{array} \right\} \sim 1\% \text{ (0.984)}$$

Also make  $10^{-4}$  soln from  $10^{-2}$  soln for AFM

SDS NT	NADDBS NT
$10^{-2} \text{ soln} = 0.0455 \text{ g} \left\{ \sim 1 \times 10^{-4} \right.$ $\text{add water till} = 4.6048 \text{ g} \left. \left( 6.95 \times 10^{-5} \right) \right\}$	$10^{-2} \text{ soln} = 0.0405 \text{ g} \left\{ \sim 1 \times 10^{-4} \right.$ $\text{add water till} = 4.0114 \text{ g} \left. \right\}$

07/18/02

NT samples I have been looking at:

<u>A</u>	<u>A1</u>	<u>E1</u>
NaDBS coated	same	1% NaDBS NT
NT	osA	spun at 6000rpm
10 <sup>4</sup> dipped, rinsed	but <u>not</u> baked	while I pipetted
<u>Baked</u>	<u>2 μm scan</u>	on <u>NT salt</u>
ddbs dep. 0.000 ← 5 μm	nadbsa1026	the Nadbsel.000
{ ddbs 10-4040 }	- .037 }	before rigorous rinsing
- .051 }		- .004
<u>4 μm scan</u>	ddbs 10-4.021	after rinsing
	↓	Nadbsel.008
	.026	- .015
	discarded double tip	same baked
		nadbsel.040
		<u>bad</u>

<u>B2</u>	<u>E</u>	<u>B</u>
1% 4000 rpm	NaDBS 1%	NaDBS NT
NaDBS salt dropped	(sample 1 month old)	1%
os chip w/	spins deposited	6000 rpm
spinning	nadbsel.000 ← before clean	spin deposited
nadbsb2.000 } before	.0017 ← after clean	<u>2 μm</u>
- .002 } cleaning	- .007 } conc. NT	{ Nadbsb.000 }
nadbsb2.003 ← after cleaning		.001 }
		image comparison
Baked	Baked	Baked
nadbsb2.040 } same conc	nadbsc.040 } same NT	
- .042 } prepared sample	.042 }	
overlapping conc. NT		

288% 70/307

158/307 ~ 60%

212/307 ~ 33%

C  
10<sup>4</sup> from 10<sup>2</sup> dipped

Nadddbs @ .001 } before baking

Nadddbs @ .040 } after baking  
.048

no tube

F  
10<sup>4</sup> from 10<sup>2</sup> Sample (D) dipped

After baking

Nadddbs f. @ .040 } no tube  
.041



cut 12 new

12-2

ddbs 12 n. 000 } Long  
.008 } tubes

ddbs 12-2.000 } not tubes

12<sup>th</sup> cut New-2

4<sup>th</sup> cut New1

20<sup>th</sup> cut

27<sup>th</sup> cut

ddbs 4 N1,000

↓

.009

Found single  
tubes

C2

dirty notube

$10^{-4}$  from  $10^{-2}$

dipped, rinsed

& baked

C3

$10^{-4}$  from  $10^{-2}$  dipped, rinsed  
baked

a lot of tubes

F2

$10^{-4}$  from  $10^{-2}$   
(sample ①)

dipped, rinsed

baked

Nddbs F2,000

↓

.009

G1

$10^{-5}$  from  $10^{-4}$

dipped, rinsed

& baked

C4

$10^{-4}$  from  $10^{-2}$  dipped,  
rinsed, baked

a lot of tubes but  
chip is dirty.

H1

H2

Bake surface  
silane  
treated, rinsed, baked

baked

same as  
H1  
not  
rinsed  
or  
baked

07/20/02

Prepare high conc. of Laser Tuber

Take 20.0g of  $5 \times 10^{-3}$  wt tubes in a glass bottle  
& slowly evaporate water to increase conc.

Empty bottle w/o cap = 13.9254 g

w/tube w/o cap 28.9274g

$\therefore$  tube wt = 15.002 g at 0.5% wt

slowly evaporate at  $44^{\circ}\text{C}$  for several days

After some solvent evaporation w/tube w/o cap = 17.4744

$\therefore$  tube soln wt = 3.549

New conc -

New conc =  $2.113 \times 10^{-2}$  wt = 2.113% by wt

Prepare 0.1% NaDBS-NT soln to sonicate 07/24/02

1% NaDBS-NT = 0.4038g }  
Add water till = 4.0127g }  $\approx 0.1\%$

Prepare 0.1% SDS-NT soln

1% SDS-NT = 0.4009g }  
Add water till = 4.0193g }